THE STUDY OF EFFECT OF CALCIUM CARBONATE SCALE PRECIPITATION TO THE CORE SAMPLE PERMEABILITY

By

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Dissertation submitted in partial fulfillment of

the Requirements for the Bachelor of Engineering (Hons)

(Petroleum Engineering)

September 2011

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CERTIFICATION OF APPROVAL

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A project dissertation submitted to the
Geosciences and Petroleum Engineering Department
Universiti Teknologi PETRONAS
in partial fulfillment of the requirement for the
Bachelor of Engineering (Hons.)
(Petroleum Engineering)

Approved by,

(Mazlin Binti Idress)

UNIVERSITI TEKNOLOGI PETRONAS
TRONOH, PERAK
September 2011

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

(MUHAMMAD IKHWAN BIN AB NASIR)

ABSTRACT

Generally, scale deposited in downhole pumps, tubing, casing flowlines, heater treaters, tanks, and other production equipment and facilities. The formations of these scales plug production lines and equipment and impair fluid flow. Their consequence could be production-equipment failure, emergency shutdown, increased maintenance cost, and an overall decrease in production efficiency. This prooject was conducted to investigate the permeability reduction caused by precipitation of calcium carbonate scale in few cores from mixing of calcium chloride solution and sodium bicarbonate solution which create artificial brine. The study will be focusing on the effect of salt concentration to the degree of scaling and to the permeability reduction of the core sample. Two experiments has been conducted which are beaker test and core test. Beaker test was conducted to determine the effect of different concentration to the amount of precipitation where the rate of reaction, nucleation and crystal growth can be observed. Core test was conducted to investigate the effect of different concentration of solution mixture to the permeability of core sample. From the result it shows a large extent of permeability damage caused by calcium carbonate that precipitated on the rock pore surface. The worst case is when 1.0M concentration of mixture used where 15% permeability decrease has been recorded when the core sample saturated in the solution. The rock permeability decline indicates the influence of the concentration of brine.

ACKNOWLEDGMENT

First and foremost I would like express my thanks to Almighty ALLAH on successful completion of this research work and thesis.

I hereby, express my sincere and profound gratitude to my supervisor Mrs Mazlin Bt Iddress for her continuing assistance, support, guidance, and understanding throughout my graduate studies. Her trust, patience, knowledge, great insight, modesty, and friendly personality have always been an inspiration for me and will deeply influence my career and future life.

The author is grateful to the Faculty of Geology and Petroleum Engineering, UTP for the support and facilities provided to carry out the experimental work. The author is also grateful to the staff of the laboratory for their support, assistance and friendly treatment that not only facilitated the work, but also made it pleasant.

My fellow Petroleum Engineering students should also be recognized for their support. Not to be forgotten, all my family members for their understanding and continuous support.

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CHAPTER 1

INTRODUCTION

1.1 Project Background

$CaCO_3$ in producing oil wells is a common problem occurs in oil and gas industry. Scale contributes to equipment corrosion and flow restriction, thus resulting in a decrease in oil and gas production. Experience in the oil industry has indicated that many oil wells have suffered flow restriction because of scale deposition within the oil producing formation matrix and the downhole equipment.

Fundamentally, such deposits appear to be due to pressure drops which allow the escape of carbon dioxide. The resulting shift in equilibrium causes the calcium carbonate to precipitate. Usually equilibrium is not completely reached so that wellhead water samples often give an indication of the tendency for scale to form in the well.

Precipitation of the mineral can occurs as fast as split of second which lead to blockage of pore throat. Blockage of the pore throat can be related with permeability reduction of the formation. Main aim of this project basically is to research on how the calcium carbonate precipitation in producing oil wells affect the permeability of the core sample which resulting to loss of production.

1.2 Oilfield Scale

The main source of oilfield scale is mixing of incompatible waters. Two waters are called incompatible if they interact chemically and precipitate minerals when mixed. A During the production, the water is drained to the surface and suffers from significant pressure drop and temperature variations. The successive pressure drops lead to release of the carbon dioxide with an increase in pH value of the produced water and precipitation of calcium carbonate (Mackay, 2003).

Common oilfield scales are:

- Calcium carbonate, $CaCO_3$
- Magnesium carbonate, $MgCO_3$
- Calcium sulphate, $CaSO_4$
- Barium sulphate, $BaSO_4$
- Strontium sulphate, $SrSO_4$
- Radium sulphate, $RaSO_4$

1.3 Scale Precipitation

Scale precipitation in surface and subsurface oil and gas production equipment has been recognized as one of the most important and serious problems that inflict oil field water injection systems. Scale limits and sometimes blocks oil and gas production by plugging the oil-producing formation matrix or fractures and perforated intervals. It can also plug production lines and equipment and impair fluid flow. The consequence could be:

i) production-equipment failure,
ii) increased maintenance cost,
iii) decrease in production efficiency.
iv) failure of these equipments could result in safety in oil and gas operation

There are other reasons why scale forms, and the amount and location of which are influenced by several factors. And, supersaturation is the most important reason behind mineral precipitation.

A supersaturated condition is the factor of scale precipitation and occurs when a solution contains dissolved materials which are at higher concentrations than their equilibrium concentration. The degree of supersaturation, also known as the scaling index, is the driving force for the precipitation reaction. It means a high supersaturation condition will lead to higher possibilities for salt precipitation.

Scale can occur at/or downstream of any point in the production system, at which supersaturation is generated. Supersaturation can be generated in single water by changing the pressure and temperature conditions or by mixing two incompatible waters. Mixing of two incompatible waters will most increase the tendency of scale precipitation.

1.4 Problem Statement

When there is appreciable quantity of scales precipitated from the brine, the fine scales agglomerate and start depositing on to a surface. Calcium carbonate can decrease the permeability in the near well area, adhere to the inside of the production tubing and clog valves and other equipment. All of these will lead to a loss in production due to:

- Formation and perforation blockage
- Scale build-up inside tubing
- Scale build-up inside pipeline
- Scale build-up in process facilities

Typical problems related to calcium carbonate are:

- Preventing movement of sliding sleeves and flow tube
- Prevent pulling of WRSCSSV
- Formation and perforation blockage
- Reduced tubing and pipeline flow area

Areas making a pressure drop such as upstream of the the downhole safet valve, crossovers, inflow valves and side pocket mandrel is exposed for calcium carbonate precipitation. Calcium carbonate is normally a problem for producers, but could also occur in high temperature injector well.

In this project, the study will be focusing on the permeability effect caused by calcium carbonate scale near wellbore area (formation).

1.5 Objectives and Scope of Study

The objectives of the research are:

1) To determine the effect of salt concentration to the degree of calcium carbonate precipitation of calcium carbonate
2) To determine the effect of different concentration to the permeability of the core sample.

The scope of study basically covers:

- A laboratory investigation of scale formation in few samples of core, resulting from the mixing of calcium chloride solution and sodium bicarbonate solution. Concentration of the solution will be the variable and will be manipulated and its effect on core permeability will be determined.
- The particle size and the morphology of scale deposition were observed using Scanning Electron Microscopy (SEM).

CHAPTER 2

LITERATURE REVIEW

2.1 The Scaling Problem in Oilfield

Scale formation is a major problem in the oil industry. They may occur downhole or in surface facilities. Generally, scale deposited in downhole pumps, tubing, casing flowlines, heater treaters, tanks, and other production equipment and facilities. The formations of these scales plug production lines and equipment and impair fluid flow. Their consequence could be production-equipment failure, emergency shutdown, increased maintenance cost, and an overall decrease in production efficiency. The failure of production equipment and instruments could lead to safety hazards.

For a scale layer to be built up, the supersaturated formation water should contact the walls of the production equipment. If the crude has a low water cut and if the water is finely dispersed in the oil, the tendency for scale to be deposited will be lowered.

The formation of inorganic mineral scale within onshore and offshore production facilities around the world is a relatively common problem. Scale can form from a single produced connate or aquifer water due to changes in temperature and pressure, or when two incompatible waters mix.

The production of such comingled fluids results in the formation of inorganic scale deposits. The types of scale and their solubility is a function of the water chemistry and physical production environment.

Oilfield scales costs are high due to intense oil and gas production decline, frequently pulling of downhole equipment for replacement, re-perforation of the producing intervals, re-drilling of plugged oil wells, stimulation of plugged oil-bearing formations, and other remedial workovers through production and injection wells. As scale deposits around the wellbore, the porous media of formation becomes plugged and may be rendered impermeable to any fluids.

It is also found downhole, deposited on the production tubing and liner resulting in reduced bore sizes and associated loss of production. Typically scale formation begins with the beginning of sea water breakthrough into a wellbore and can lead to very rapid production declines.

2.2 Inorganic Scale

Inorganic scales are mineral deposits (salt deposits) from aqueous solutions of minerals (brines) due to disturbance in thermodynamic and chemical equilibrium resulting in supersaturation. One major group of inorganic scales is carbonate and sulphate scales, where one of the alkaline earth metals Mg, Ca, Sr, Ba or Ra are present. When dissolved in the water, the carbonate and sulphate are present as negative ions, while the alkaline earth metals are present as positive ions. Common scale types within this group are:

- Calcium carbonate, $CaCO_3$
- Magnesium carbonate, $MgCO_3$
- Calcium sulphate, $CaSO_4$
- Barium sulphate, $BaSO_4$
- Strontium sulphate, $SrSO_4$
- Radium sulphate, $RaSO_4$

The above carbonate deposits depend on changing operational conditions such as pressure and temperature. Sulphate scale deposits depend on mixing incompatible waters, such as formation water rich in Ca, Sr and Ba, and sea water rich in sulphate. Sulphate scale deposits will often be a mixture of $CaSO_4$, $BaSO_4$, and $SrSO_4$. $RaSO_4$ is radioacive and may also be present in the sulphate scale mixture. The most predominant scale types in the above list are calcium carbonate and barium sulphate.

Other inorganic scale is:

- Sodium chloride, NaCl

Sodium chloride deposits depend on changing operational conditions such as pressure and temperature. Note that all salt deposits described above forms in water and can only occur when water is contacting a surface.Another group of inorganic scales are the iron scales including:

- Iron carbonate, $FeCO_3$ (Siderite)
- Iron sulphide, FeS (Trolite)

The iron above is often taken from the well materials as a result of corrosion processes. Iron carbonate is common when having CO_2 corrosion. The ion sulfide (FeS) is a black solid that may be produced by sulphate reducing bacteria (SRB) in the reaction:

$$4Fe + SO_4^{2-} + 4H_2O \rightarrow 3Fe(OH)_2 + FeS + 2OH^-$$

Iron oxides as corrosion products (rust) are also defined as inorganic scales in wells. Common types of iron oxides are:

- Fe_2O_3 (Hematite)
- Fe_3O_4 (Magnetite)

2.3 Calcium Carbonate ($CaCO_3$) Scale

Two main types of scale which are commonly found in the oilfield are carbonate and sulfate scales. The formation of carbonate scale is associated with the pressure, temperature, total dissolve salt and pH changes of the production fluid.

2.3.1 Calcium carbonate ($CaCO_3$) scale precipitation mechanism

Calcium carbonate or calcite scale is frequently encountered in oilfield operations. But the calcite has the greatest stability in oilfield circumstances, so it is the most common form of calcium carbonate encountered in oilfield production operation.

Deposition of $CaCO_3$ scale results from precipitation of calcium carbonate is as per following equation:

$$Ca^{2+} + CO_3^{2-} \rightarrow CaCO_3$$

Calcium carbonate scale can also be formed by combination of calcium and bicarbonate ions, and this reaction is the major cause of calcium carbonate scale deposition in oilfield operations.

In many oilfields, the deposition of calcium carbonate scale on surface and subsurface production equipment creates an operation problem. It occurs when the formation water becomes supersaturated with calcium carbonate because of the drop in pressure during production. The continuous flow of a supersaturated solution through the production equipment has lead to the precipitation the calcium carbonate layer.

Carbonate scales frequently appear in the wellbore, especially near the wellhead where, because of pressure drop, dissolved CO_2 escaped from produced water and caused water pH as well as the saturation index of carbonate minerals to increase (Zhang and Farquhar, 2001).

In the pre-seawater breakthrough period, calcium carbonate precipitation, caused by the loss of CO_2 from the formation water produced, can be observed. $CaCO_3$ scaling is not difficult to control by scale inhibitors or by removal with acid.

During the production, the water is drained to the surface and suffers from significant pressure drop and temperature variations during the production. The continuous pressure drops lead to degassing of the carbon dioxide with an increase in pH value of the produced water and precipitation of calcium carbonate (Mackay, 2003; Rousseau *et al.*, 2003).

Carbonate scale formation occurs when connate water or aquifer water passes through the bubble point and carbon dioxide is evolved. As carbon dioxide is evolved, the solubility with respect to carbonate declines rapidly and forms a precipitate with divalent ions, such as iron, and more commonly calcium, as outlined in the following equation (Mackay and Jordan, 2005):

$$Ca\,(HCO_3) \Leftrightarrow CaCO_3 + CO_2 + H_2O$$

According to Clemmit *et al.*, (1985), calcium carbonate scale is formed by a different mechanism. As few waters contain the actual carbonate ion, the scaling potential arises from decomposition of calcium bicarbonate. This decomposition is due to the pressure reductions (at chokes or separators) releasing carbon dioxide and thus moving the equilibrium (with a pH increase) of the above reaction to the right and producing calcium carbonate. If the quantity of calcium carbonate produced exceeds its solubility in the water, then precipitation can occur.

2.3.2 Supersaturation

The saturation ratio, SR, is defined as:

$$SR = \frac{a_{Ca^{2+}} a_{CO_3^{2-}}}{K_{SP}^{a}} = \frac{m_{Ca^{2+}} m_{CO_3^{2-}}}{K_{SP}}$$

a and γ are the activity and activity coefficient, respectively and **m**$_i$ the molality of ion i. The classification can be summarized as:

i) If SR<1, the system is undersaturated.
ii) If SR=1, the system is saturated while
iii) If SR>1, the system is supersaturated.

Supersaturation is the most important reason behind mineral precipitation. A supersaturated is the primary cause of scale formation and occurs when a solution contains dissolved materials which are at higher concentrations than their equilibrium concentration. The degree of supersaturation, also known as the scaling index, is the driving force for the precipitation reaction and a high supersaturation, therefore, implies high possibilities for salt precipitation.

Supersaturation exists in a metastable state and, as such, the manner in which it exists in solution or comes out of solution by crystallization and precipitation is entirely unpredictable.

2.3.3 Calcium carbonate ($CaCO_3$) scale precipitation influencing factors

Required environmental conditions are:

- Formation water
- Reduced saturation limit for dissolved ions

The components for calcium carbonate are calcium (Ca^{2+}), bicarbonate (HCO_3^-) and/or carbonate (CO_3^{2-}). These components are normal in formation water and will precipitate due to reduced saturation limit for the ions in the water. The saturation limit for dissolved ions is reduced by reduced pressure, increased temperature, increased pH and reduced ionic strength.

The effect of reduced pressure is often dominant in production wells. Injection wells can have scale due to increasing temperature downhole. More about influencing factors follows:

i) Formation water and Salt concentration

Water is a good solvent for many materials and will become rich in ions from several minerals. Water located in carbonate and calcite cemented sandstone reservoirs usually contains high levels of calcium ions which increases the risk for calcium carbonate deposits.

ii) Reduced pressure

The saturation limit for dissolved ions reduces with reduced pressure causing increased $CaCO_3$precipitation at reduced pressure:

$$Ca^{2+} (aq) + CO_3^{2-} (aq) \rightarrow CaCO_3 (s)$$

The reason is reduced amount of CO_2 dissolved in the water as the pressure is reduced.

$$H^+ + HCO_3^- \rightarrow H_2CO_3 \rightarrow H_2O + CO_2 (aq)$$

$$CO_2 (aq) \rightarrow CO_2 (g)$$

The reduction in H^+ ions means increased pH, causing decreased solubility of dissolved carbonate ions (CO_3^{2-}), causing increased precipitation of $CaCO_3$. The free CO_2 gas goes to the oil phase.

Calcium carbonate scale is normally not a problem in the near wellbore area and in the bottom of producing wells due to low pressure drop. Calcium carbonate scale precipitation and deposition may occur higher up in the tubing where the pressure has been reduced, like near the safety valve and the wellhead. The area after the choke is critical due to high pressure drop.

The potential for carbonate scale formation is highest at water breakthrough. The combination of low water cut < 1% and high pressure drop is especially critical. The reservoir pressure will gradually decline from production start up to shut down. The reservoir pressure is normally supported by injection wells some years but not the entire lifetime of the well. The pressure may be reduced with several hundred bars in some high pressure (HP) wells.

Calcium carbonate may be a problem also in the near well bore area and in the production tubing when combining relative high reservoir temperature with high pressure drops in the later phase of the production period. In injection wells, scale may occur anywhere. Pressure profiles in the flow path for different scenario during the lifetime of the well are important as an input parameter during the calcium carbonate assessment.

iii) Increased temperature

The saturation limit for dissolved ions reduces with increased temperature causing increased $CaCO_3$ precipitation at higher temperature.

$$Ca^{2+}\,(aq) + CO_3^{2-}\,(aq) \rightarrow CaCO_3\,(s)$$

The reason is reduced amount of CO_2 dissolved in the water as the temperature increases. This will increase the pH of the solution as explained above. The amount of CO_3^{2-} ions will increase with increased pH causing increased $CaCO_3$ precipitation. Temperature profiles in the flow path for different scenario during the lifetime of the well are important as an input parameter during the calcium carbonate assessment.

iv) Increased pH

Increased pH will reduce the solubility of salt and thus increase scale deposits. The presence of CO_2 gas and water make carbonic acid (H_2CO_3). The carbonic acid will further dissociate to H^+ and HCO_3^- ions (bicarbonate). The H^+ ions will reduce the pH in the solution and thus reduce the $CaCO_3$ precipitation. Note the effect the pressure and temperature have on the carbonic acid strength as described above.

Presence of organic acids (reduced pH) in the formation water will also affect the pH and hence the solubility of calcium carbonate. Presence of organic acids will normally not eliminate scaling, but the scaling potential may be reduced.

v) Reduced ionic strength

Ionic strength of a solution is a function of the concentration of all ions present in the solution. Increased concentration of e.g. Na^+ and Cl^- will increase the ionic strength. Increased ionic strength has the effect that more CO_3^{2-} and Ca^{2+} ions can be dissolved without making $CaCO_3$.

Increased ionic activity from foreign ions will shield the calcium and the carbonate ion from each other and reduce their possibility to make scale. Thus, reduced ionic strength will increase the probability for having $CaCO_3$.

2.4 Permeability reduction

From the core flood test, the flow rate across the core was recorded continuously and the permeability of core was calculated using Darcy's linear-flow equation. The decrease in flow rate occurred when supersaturated brine was flowing through the cores. This indicates that the decrease is due to precipitation of the mineral or salt in the core with the consequent reduction in its permeability and porosity.

The reduction in permeability is possibly caused by precipitation crystal has blocked the pore throats which can be observed by the Scanning Electron Microscopy, SEM. As concentration of brine (i.e. supersaturation) is increasing, permeability loss occurs more rapidly.

Darcy's Law: A proportional relationship between the instantaneous discharge rate through a porous medium, the viscosity of the fluid and the pressure drop over a given distance, L, which is:

$$Q = \frac{-\kappa A}{\mu} \frac{(P_b - P_a)}{L}$$

Where Q: production flow rate

k: permeability

μ: viscosity

L: distance

P_b: downstream pressure

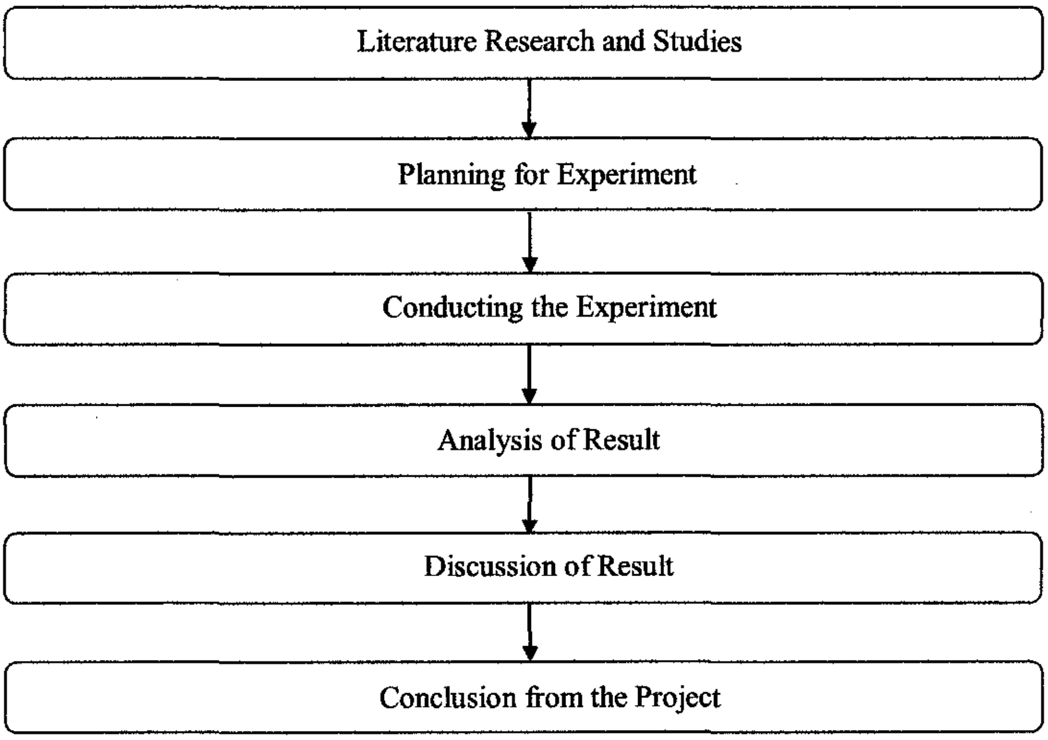
P_a: upstream pressure

CHAPTER 3

METHODOLOGY

3.1 Workflow of Project

Several main procedures have been identified towards accomplishment of the project. The following diagram summarizes on the tasks to be perform accordingly.



First step during the kick start of the project would be literature review and research by author through journals, websites, textbooks, SPE papers and Google.

3.2 Material Used

The materials used in this study were:

3.2.1 Salt Solution

Calcium carbonate will be precipitated by existence of Ca^{2+} ion and CO_3^{2-} ion. Hence, two solutions have to be prepared which are:

i) Calcium chloride solution
ii) Sodium carbonate solution

The mixture of these two solutions will definitely precipitate calcium carbonate scale and the mixture can be assumed as artificial brine.

3.2.2 Core Sample

In all flooding experiments, cores from random type being used. 5 core samples with 3 inch length and 1 inch diameter are being prepared. No oil was present in the cores.

Table 3.1: Parameter of core sample

Core sample	Length (inch)	Diameter (inch)	Permeability (md)
1	3	1	
2	3	1	
3	3	1	
4	3	1	
5	3	1	

3.3 Equipment Set up

A schematic diagram and photograph of the experimental set-up used in this study were shown in figure below. It comprised of:

i) Scaling test rig for coreflood test

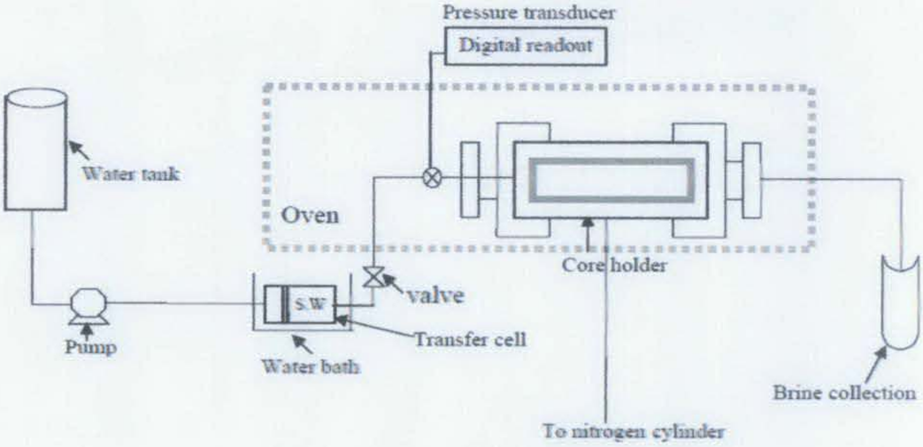


Figure 3.1: Schematic diagram of coreflooding equipment

ii) Scanning Electron Microscopic (SEM) to observe the precipitates morphology

Figure 3.2: SEM

3.4 Experimental Procedure

There are two purposes of experimental study which are:

i) to investigate the effect of different solution concentrations on the precipitation of calcium carbonate

ii) to investigate permeability reduction by precipitation of scale in a core sample

3.4.1 Beaker Test (Crystallization experiments)

The intention of this study is to determine the effect of different concentration to the amount of precipitation. The experimental procedures used in the determination of concentration effect were:

a) For 0.1M $CaCO_3$

i) A growth solution of 0.1M $CaCO_3$ was prepared by mixing 200ml $CaCl_2$ (0.2M) and 200ml Na_2CO_3 (0.2M) each in a beaker
ii) Stir the mixture solution using stirrer.
iii) After 15 minutes, pour 50 ml of the mixture into another beaker and then filter the mixture using filter paper and funnel. The crystal filtered then dried for 24 hours.
iv) Repeat step (iii) until a sample produced for every 15 minutes. 6 samples will be produced in the end.
v) The weight of the crystals was measured and the amount of precipitated was calculated.

b) For 0.3M $CaCO_3$

iii) A growth solution of 0.3M $CaCO_3$ was prepared by mixing 200ml $CaCl_2$ (0.6M) and 200ml Na_2CO_3 (0.6M) each in a beaker

iv) Stir the mixture solution using stirrer.

v) After 15 minutes, pour 50 ml of the mixture into another beaker and then filter the mixture using filter paper and funnel. The crystal filtered then dried for 24 hours.

vi) Repeat step (iii) until a sample produced for every 15 minutes. 6 samples will be produced in the end.

vii) The weight of the crystals was measured and the amount of precipitated was calculated.

c) For 0.6M $CaCO_3$

i) A growth solution of 0.6M $CaCO_3$ was prepared by mixing 200ml $CaCl_2$ (1.2M) and 200ml Na_2CO_3 (1.2M) each in a beaker

ii) Stir the mixture solution using stirrer.

iii) After 15 minutes, pour 50 ml of the mixture into another beaker and then filter the mixture using filter paper and funnel. The crystal filtered then dried for 24 hours.

iv) Repeat step (iii) until a sample produced for every 15 minutes. 6 samples will be produced in the end.

v) The weight of the crystals was measured and the amount of precipitated was calculated.

d) For 0.8M $CaCO_3$

i) A growth solution of 0.8M $CaCO_3$ was prepared by mixing 200ml $CaCl_2$ (1.6M) and 200ml Na_2CO_3 (1.6M) each in a beaker

ii) Stir the mixture solution using stirrer.

iii) After 15 minutes, pour 50 ml of the mixture into another beaker and then filter the mixture using filter paper and funnel. The crystal filtered then dried for 24 hours.

iv) Repeat step (iii) until a sample produced for every 15 minutes. 6 samples will be produced in the end.

v) The weight of the crystals was measured and the amount of precipitated was calculated.

e) For 1.0M $CaCO_3$

i) A growth solution of 1.0M $CaCO_3$ was prepared by mixing 200ml $CaCl_2$ (2.0M) and 200ml Na_2CO_3 (2.0M) each in a beaker

ii) Stir the mixture solution using stirrer.

iii) After 15 minutes, pour 50 ml of the mixture into another beaker and then filter the mixture using filter paper and funnel. The crystal filtered then dried for 24 hours.

iv) Repeat step (iii) until a sample produced for every 15 minutes. 6 samples will be produced in the end.

v) The weight of the crystals was measured and the amount of precipitated was calculated.

3.4.2 Core Test

The test rig was designed to investigate the effect of different concentration to the permeability of core sample.

3.4.2.1 Core preparation

i) Before each run, the core was dried at room temperature for 24 hours.

3.4.2.2 Initial porosity and permeability measurement

i) Measure the initial porosity and permeability of the core using POROPERM. POROPERM is an apparatus for measuring the permeability of a core sample. Measurements are made either by placing the sample in a chamber (also known as a core holder), or by placing a probe on the surface of the sample.

Figure 3.3: POROPERM machine

3.4.2.3 Flooding experiment

The procedure of flooding experiment is as given below:

i) The system consisting of the core holder assembly with the core sample placed inside the oven and transfer cell containing sea water was then placed inside the water bath and heated to the desired temperature of the run. The required confining pressure was then adjusted to be approximately at double inlet pressure.

ii) A flooding run was started by setting plunger pump at different pressures. Thus, the artificial brine (mixture of calcium chloride and sodium carbonate solution) was injected into the core. The inlet pressure was measured by pressure transducer while the outlet pressure was atmospheric pressure.

iii) During each run, the flow rate across the core was recorded continuously and the permeability of core was recorded.

iv) The core sample was removed at the end of flooding then dried and cut into sections for Scanning Electron Microscopy (SEM).

3.4.2.4 Scanning Electron Microscopy (SEM)

Scanning Electron Microscopy (SEM) was used to examine the cores before and after scale deposition. The core sample was removed at the end of flooding and broken into sections. The front of the cores was then examined by SEM to reveal the morphology of scale formation crystals

3.5 Project Planning – Gantt Chart for FYP II

No	Detail / Week	1	2	3	4	5	6	7		8	9	10	11	12	13	14	15	16
1	Briefing & update on student progress			■					Mid Semester Break									
2	Project work commences	■	■	■	■	■	■	■		■	■	■	■	■				
3	Submission of Progress Report									■								
4	PRE-EDX, Poster Presentation, Submission of Final Report (CD Softcopy & Softbound)												■					
5	EDX													■				
6	Final Oral Presentation														■			
7	Delivery of Final Report to External Examiner / Marking by External Examiner															■		
8	Submission of Hardbound Copies																	■

CHAPTER 4

RESULTS AND FINDINGS

In this chapter, the progress, the experimental results and the findings up to this time being are going to be presented.

As described in the previous chapter, the experimental part of this project can be divided into two parts which are:

i) Beaker test

ii) Core test

4.1 Beaker Test

For the beaker test which objectively aims to investigate the effect of solution concentration on the amount of calcium carbonate precipitation, the experiment has been conducted as per experimental procedure.

4.1.1 Data Analysis

Data were obtained as the amount of precipitate versus time at various concentrations of the growth solution and each data was plotted as the amount of precipitate versus time using Microsoft Excel 2007

From the experiment conducted, result can be presented as below:

a) For 0.1M $CaCO_3$

Table 4.1: Result of 0.1M $CaCO_3$ concentration Experiment

Time	Weight of crystals (g)	Amount of precipitation (g/L)
0	0.1	2
15	0.104	2.08
30	0.106	2.12
45	0.1085	2.17
60	0.1125	2.25
75	0.113	2.26
90	0.113	2.26

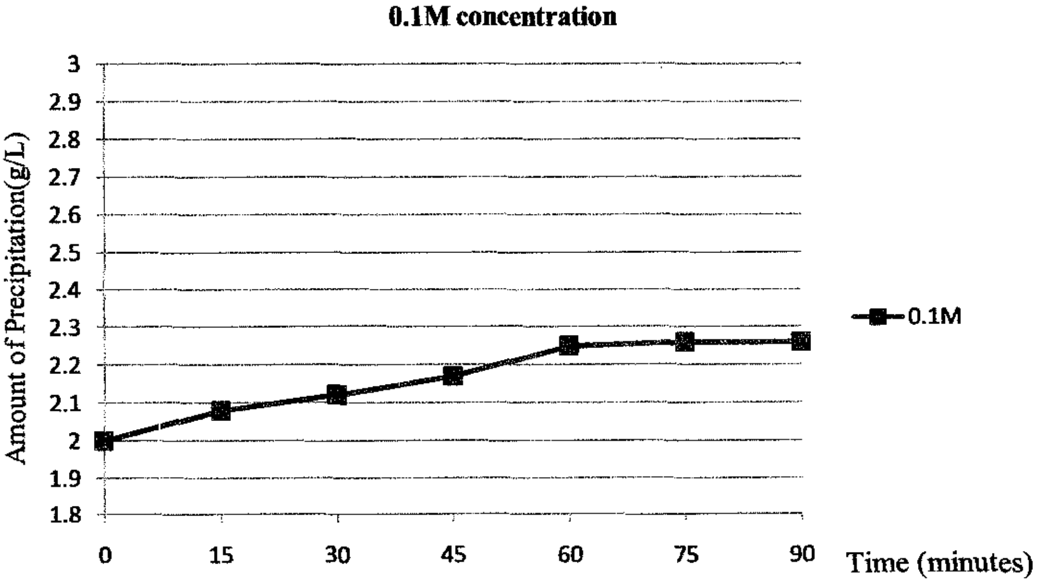


Figure 4.1: Plotted based on Result of 0.1M $CaCO_3$ concentration Experiment

The result when 0.1M concentration being used has been tabulate in the table and a graph has been plotted based on the result. The trend of increasing rate of reaction can be observed from this result.

b) **For 0.3M $CaCO_3$**

Table 4.2: Result of 0.3M $CaCO_3$ concentration Experiment

Time	Weight of crystals (g)	Amount of precipitation (g/L)
0	0.1	2
15	0.106	2.12
30	0.114	2.28
45	0.1205	2.41
60	0.121	2.42
75	0.1214	2.428
90	0.1218	2.436

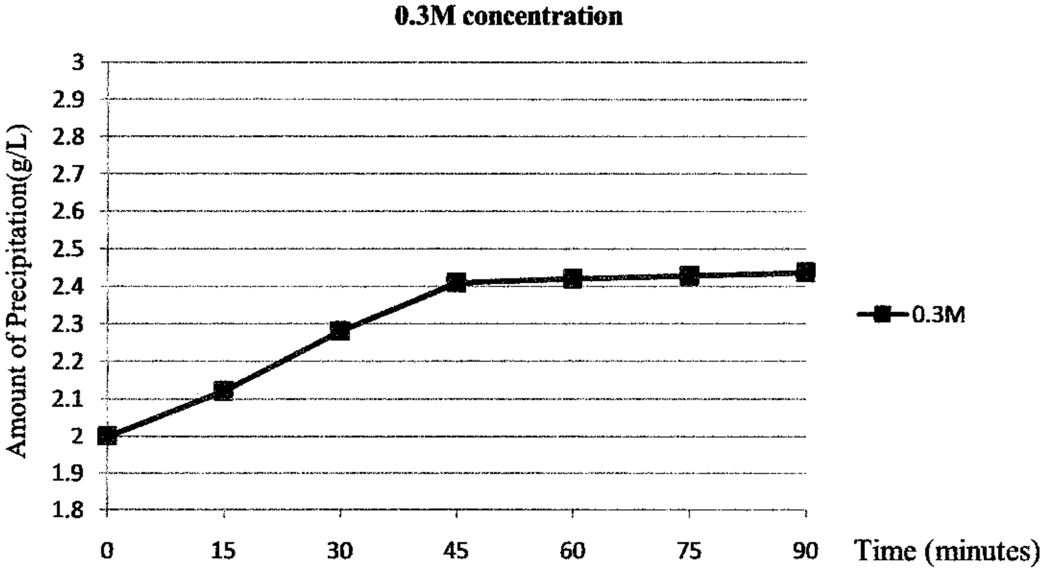


Figure 4.2: Plotted based on Result of 0.3M $CaCO_3$ concentration Experiment

The result when 0.3M concentration being used has been tabulate in the table and a graph has been plotted based on the result. The trend of increasing rate of reaction can be observed from this result as well.

c) For 0.6M $CaCO_3$

Table 4.3: Result of 0.6M $CaCO_3$ concentration Experiment

Time	Weight of crystals (g)	Amount of precipitation (g/L)
0	0.1	2
15	0.114	2.28
30	0.1225	2.45
45	0.1295	2.59
60	0.1294	2.588
75	0.1297	2.594
90	0.1298	2.596

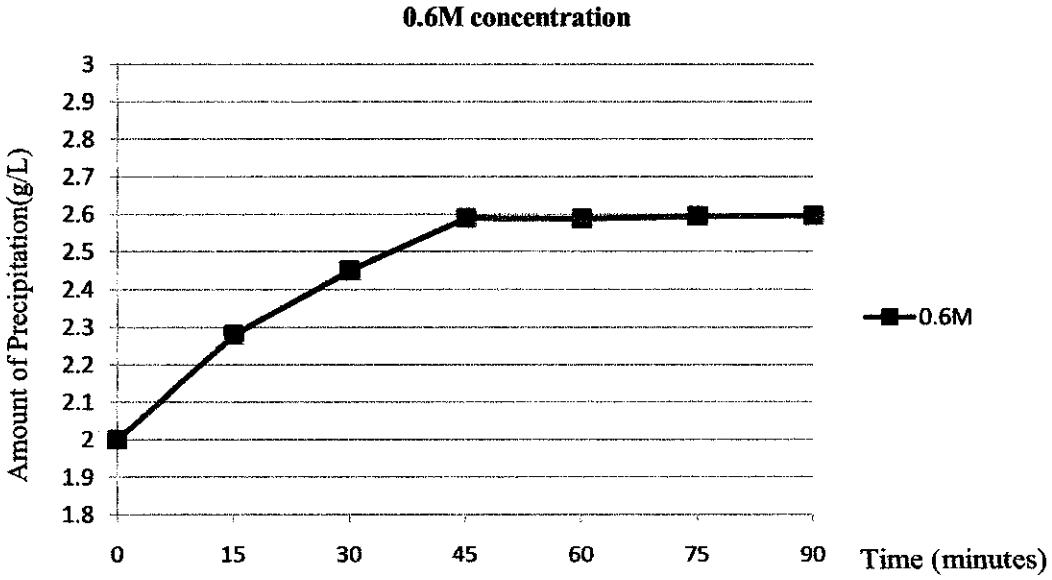


Figure 4.3: Plotted based on Result of 0.6M $CaCO_3$ concentration Experiment

The result when 0.6M concentration being used has been tabulate in the table and a graph has been plotted based on the result. The trend of increasing rate of reaction can be observed from this result. The reaction also observed to stop at 45 minutes.

d) For 0.8M $CaCO_3$

Table 4.4: Result of 0.8M $CaCO_3$ concentration Experiment

Time	Weight of crystals (g)	Amount of precipitation (g/L)
0	0.1	2
15	0.1175	2.35
30	0.126	2.52
45	0.135	2.7
60	0.136	2.72
75	0.134	2.68
90	0.135	2.7

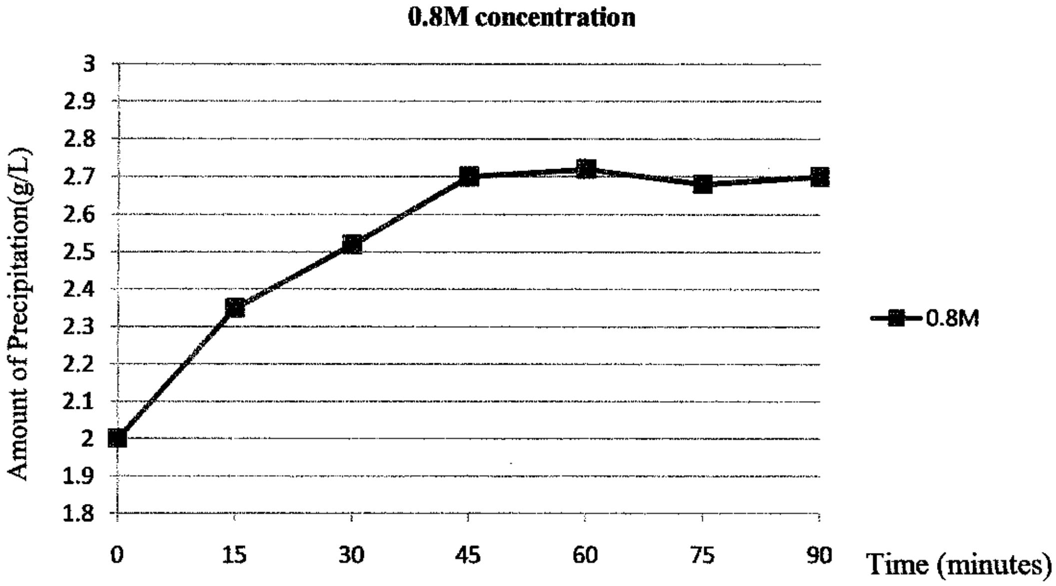


Figure 4.4: Plotted based on Result of 0.8M $CaCO_3$ concentration Experiment

The result when 0.8M concentration being used has been tabulate in the table and a graph has been plotted based on the result. The trend of increasing rate of reaction can be observed from this result. The reaction also observed to stop at 45 minutes.

e) For 1.0M $CaCO_3$

Table 4.5: Result of 1.0M $CaCO_3$ concentration Experiment

Time	Weight of crystals (g)	Amount of precipitation (g/L)
0	0.1	2
15	0.1245	2.49
30	0.13	2.6
45	0.14	2.8
60	0.141	2.82
75	0.143	2.86
90	0.144	2.88

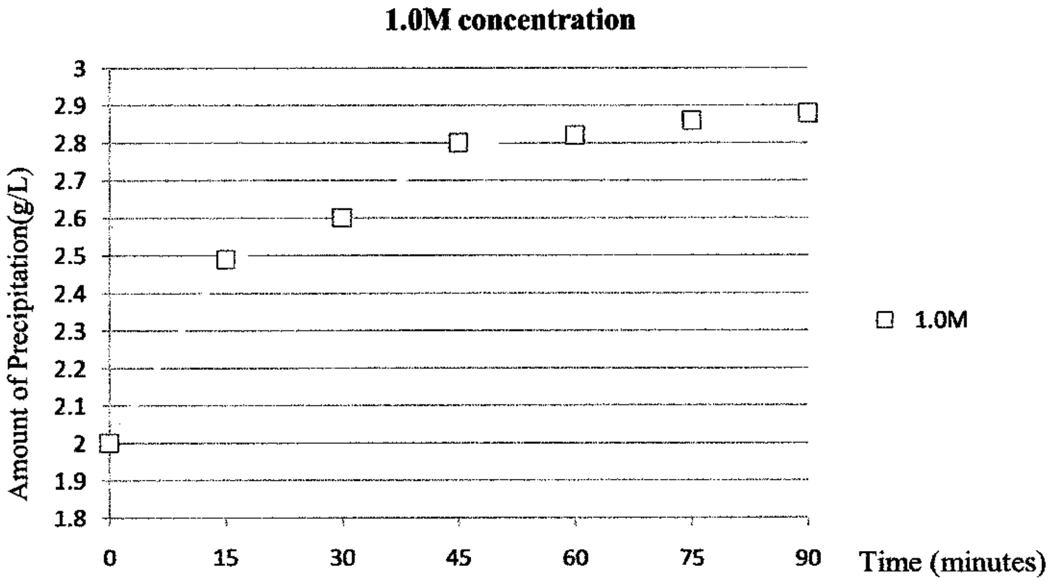


Figure 4.5: Plotted based on Result of 1.0M $CaCO_3$ concentration Experiment

The result when 1.0M concentration being used has been tabulate in the table and a graph has been plotted based on the result. The trend of increasing rate of reaction can be observed from this result. The reaction also observed to stop at 45 minutes.

f) Comparison of different solution concentration and its effect to amount of precipitation

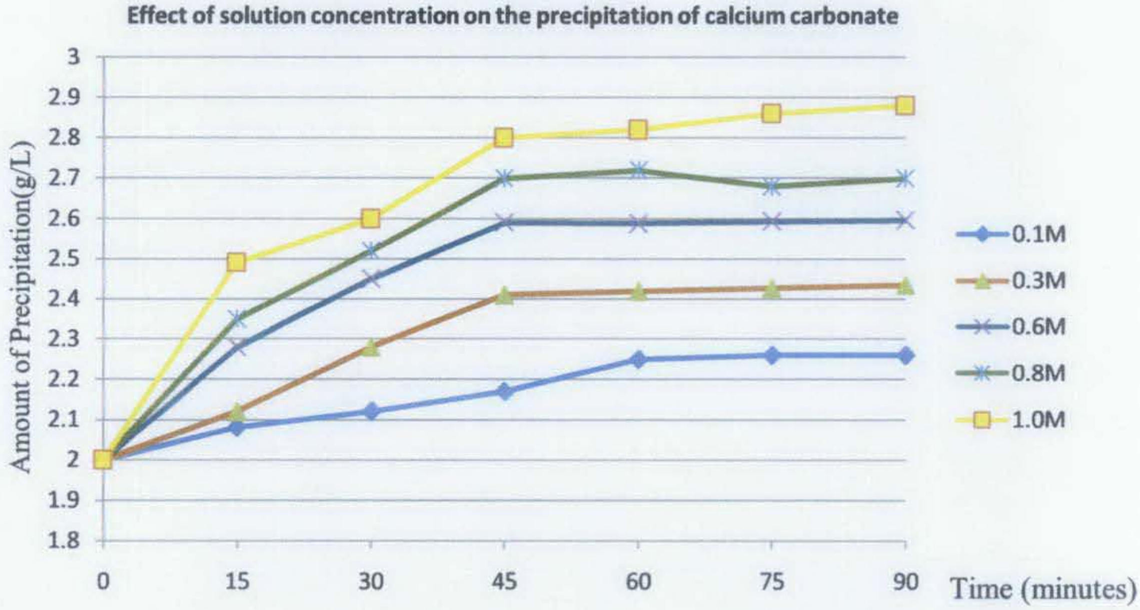


Figure 4.6: Comparison of the experiment result using five different concentrations

From this experiment, it shows that the amount of precipitated $CaCO_3$ increase from time 0 to 90 minutes. As per result obtained in the end of the experiment, it is shown that the higher the concentration of salt solution, the higher the amount of calcium carbonate precipitation.

Based on the graph plotted based on the result, it is shown that the precipitation occurs rapidly at the early minutes of the experiment. Starting from the half way of the experiment until the end, the mineral precipitation seems a little bit slow and compared to the early stage of the experiment. This phenomenon might occur because the precipitation reaction has reached its equilibrium.

4.2 Core Test

The main objective of this part of the investigation is to study permeability reduction caused by calcium carbonate scale deposition in core sample. The core test was designed to investigate the effect of calcium carbonate concentration to the permeability reduction. During each run, permeability of the core before and after the saturation has been recorded. It was observed the permeability reduced after the saturation process.

4.2.1 Limitation

The plan to conduct core test using the scaling test rig for coreflood test (refer: Figure 1) has been diverted due to unavailability of the equipment due to long queue of booking. Alternatively, saturation process has to be conducted by flooding the core in the solution manually in room condition.

The initial plan to use Scanning Electron Microscopy (SEM) to examine the sample morphology before and after scale precipitation also has to be diverted due to the time limitation. Alternatively, illustration will be provided to relate the effect of concentration to the permeability reduction

4.2.2 Results and findings

Table 4.6: Result of Core Test Experiment

Sample	Concentration (M)	Initial Permeability (md)	Final Permeability (md)	Permeability Reduction (%)
1	0.1	119	114	4.2
2	0.3	135	126	6.7
3	0.6	121	110	9.1
4	0.8	128	111	13.3
5	1.0	140	119	15.0

Table 4.6 shows the variation in permeability decline for different concentrations of calcium carbonate. These table shows that the effect of concentration on permeability reduction. An increment in concentration of salt has been observed to increase the supersaturation of brine which leads to the increasing rate of nucleation and crystal growth as per crystallization experiment in the first part. Therefore it can be summarized that increment of concentration will cause the rate of reaction, nucleation, crystal growth and plugging to increase.

The increase of the rate of reaction, nucleation, crystal growth and plugging will increase the degree of severity of the permeability reduction of the sample. The interconnected pore spaces area will decrease due to the precipitation of the calcium carbonate mineral. The decrease of interconnected pore spaces area has caused the permeability to reduce drastically.

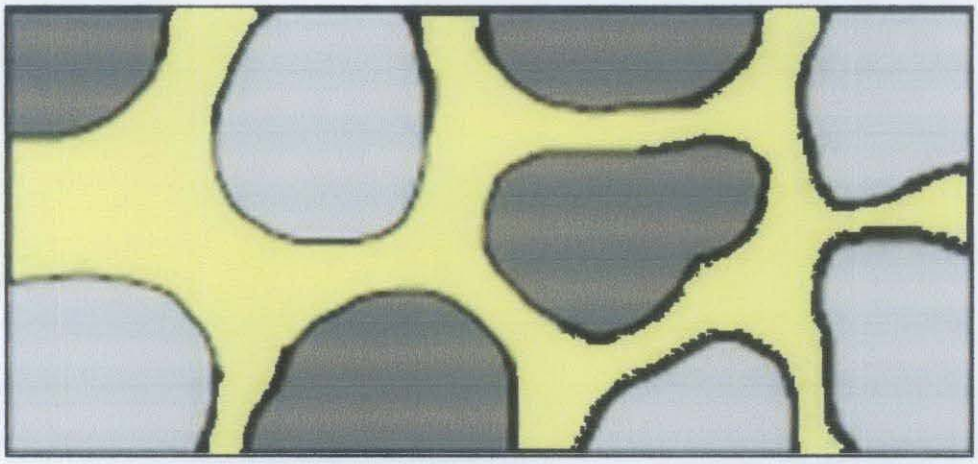


Figure 4.7: Interconnected pore spaces before mineral precipitation

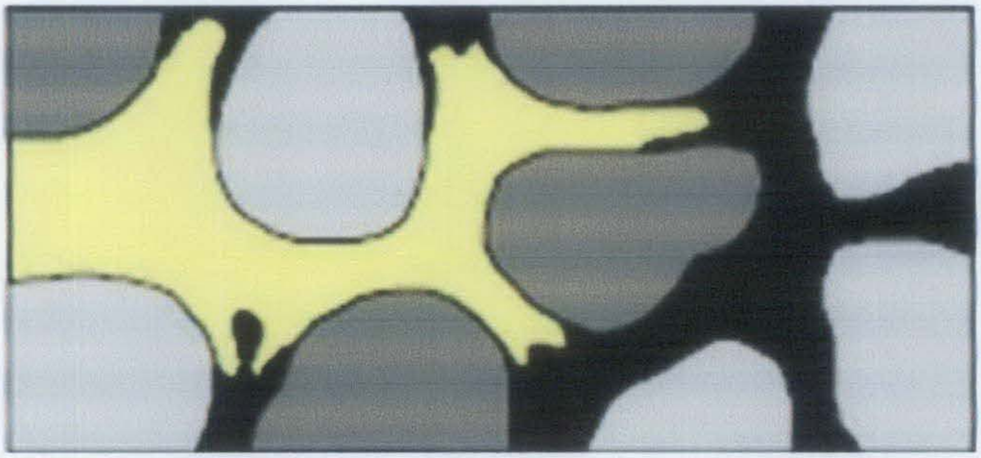


Figure 4.8: Mineral precipitation inside interconnected pore spaces

The illustration on the significant of calcium carbonate precipitation to the permeability reduction can be summarized in Figure 4.7 and Figure 4.8 where it is clearly shown that the precipitation has decrease the interconnected pore spaces area. Thus, the reduction in permeability is occurred due crystals blocking the pore throats.

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

The work carried out in this study focused on the effect concentration of salt solution on permeability reduction by deposition of scale formation in a porous medium.

Beaker test which conducted with various solution concentrations gave the following conclusions.

(1) The beaker test results confirm the general trend in the rate of reaction, nucleation and crystal growth increase rapidly when the concentrations of salt mixture increase.

(2) The increase of the rate of reaction, nucleation and crystal growth has lead to the larger amount of calcium carbonate mineral precipitation.

(3) The rate of reaction, nucleation and crystal growth of calcium carbonate occurs rapidly but it reaches equilibrium rapidly as well. It is proven from the experiment when the reaction stopped at 45 minutes after the experiment being conducted. It shows that calcium carbonate precipitation can occurs in a very large amount within

few seconds in oilfield and it takes just a few minutes to stop the production of a well if there is no prevention being taken.

For the core test which is conducted in consequence of the beaker test, various concentrations of salt mixture being flooded and saturated into the core sample and gave following conclusions.

(1) The core test results confirm the general trend in concentration dependencies for the permeability reduction of core sample. An increase in solution concentration has lead to higher percentage of permeability reduction. It is proven when 0.1M concentration being used, only 4.2% permeability reduction occurred while 1.0M concentration caused 15% permeability reduction.

(2) Higher concentration of salt which previously known to increase the rate of reaction, nucleation and crystal growth of calcium carbonate has plugged the interconnected pore spaces area in core sample.

(3) Higher concentration of salt precipitate larger amount of calcium carbonate which decrease larger area of interconnected pore spaces in core. Decreasing of interconnected pore spaces in core will reduce the permeability of core sample.

(4) It confirms that salt concentration is one of the factors which play a very important role in determining the degree of severity of calcium carbonate scale precipitation.

5.2 Recommendations

Based on the results and conclusions obtained from this study, the following suggestions for future work in the same area are recommended:

(1) Instead of salt solution being used, real oil field brines can be employed in the study by mixing field disposal water and seawater.

(2) Improvement could be done by performing morphology test using Scanning Electron Microscopy (SEM) to check the pore spaces plugging.

(3) Saturation process should be conducted using coreflood equipment rather than employing manual saturation method which produces less accurate result.

(4) The concentrations of calcium chloride and sodium carbonate solution could be determined and much improved using an in–line ion analyzer or some other analytical devices rather than using manual calculation method which can produce more accurate result.

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